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FSTC-HT-23-490-69

U.S. ARMY FOREIGN SCIENCE AND TECHNOLOGY CENTER



INVESTIGATIONS INTO THE PRIMARY PRODUCTS OF IMPULSE PARALYSIS OF
AMMONIUM PERCHLORATE USING A TIME OF FLIGHT MASS SPECTROMETER

COUNTRY: USSR

TECHNICAL TRANSLATION

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by

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This material has universal decimal classification
542.92 + 546.137 + 546.39

Source: IZVESTIYA AKADEMII NAUK SSSR
SERIYA KHIMICHESKAYA
(News of the Inventions Academy
Chemical Series)
No. 6, 1968
USSR

Translated for FSTC by Techtran Corporation

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INVESTIGATIONS INTO THE PRIMARY PRODUCTS OF IMPULSE PARALYSIS OF AMMONIUM PERCHLORATE USING A TIME OF FLIGHT MASS SPECTROMETER

In studying the processes of thermal breakdown in solid substances, it is especially important to examine the mechanism in the first stages of the process, which is frequently masked by secondary reactions. To solve this problem in the investigation of the thermal dissolution mechanism for ammonium perchlorate in previous works /1, 2/ we used the method of impulse heating for a solid substance in the ion source of a time of flight mass spectrometer. This method permits the study only of stable products of the breakdown of solid substances -- atoms, radicals, and gases which easily react under ordinary conditions with metals, -- cannot be observed using this device, if their time of existence is less than the time from their moment of formation until the moment of registration in the mass spectrometer.

This condition was evidently the reason why, in /1, 2/ only one of the products of the thermal breakdown of ammonium perchlorate was observed, ammonia. The other theoretical product of thermal breakdown -- perchloric acid, was not registered, apparently due to its instability. This could be, for example, if the time of its destruction in the chamber of the mass spectrometer sensing unit was less than 0.05 seconds -- a constant time for the registration of mass spectra on movie film from the screen of an oscillograph in the experimental method used by us.

Experimental portion

To eliminate the difficulties described, the method used in /1, 2/ was modified, so that the surface of the titanium heater strip on which the crystals of ammonium perchlorate were placed, was parallel to the first net of the ion source and faced the side of this net, a few millimeters away from it. When the crystals were impulse heated to 400° the breakdown lasted for several milliseconds. During this period the time from the beginning of the reaction to the moment of registration was one millisecond. In Fig. 1 we show the position of the titanium strip with the suspended solid substance in the ion source. The products of reaction escape from the surface of the titanium strip in the direction of the ion source. The dotted lines show the path of these fragments, which pass into the ionization space before their first collision with the walls of the sensing apparatus, where the active fragments can be destroyed. It must be remarked that in this case the intensity of the lines in the mass spectra is proportional to the pressure in the stream of breakdown products, which is proportional to the density of the reaction products from the surface of the specimen, that is, the speed of breakdown of the solid substance.

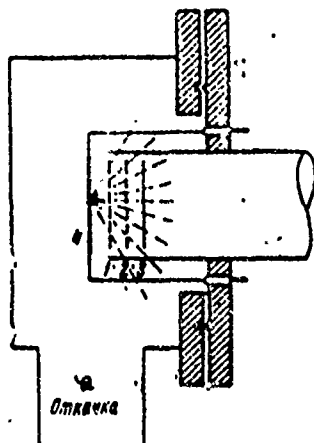


Fig. 1. Position of the heater with the mounting in the ion source: 1, 2, 3 -- screens of the ion source; 4. titanium strip

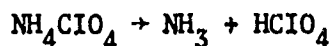
Key: a. exhaust

The table below gives the relative intensities of the lines in the mass spectra of the stream of breakdown products from ammonium perchlorate at 400° (the energy of the ionizing electrons was 70 electron volts) one millisecond after the start of the reaction.

a m/e, ion	16, NH ₄ ⁺	17, NH ₃ ⁺ , OH ⁺	35, Cl ⁺	51, ClO ⁺	67, ClO ₂ ⁺	83, ClO ₃ ⁺	100, HClO ₄ ⁺
b Интенсивность, %	53	93	13	13	73	100	54

Key: a. m/e, ion; b. Intensity, %

The ratio of the intensities of masses at 35, 51, 67, 83 and 100 are close to the ratios of these masses in the mass spectrum of perchloric acid, obtained by Fisher /3/ in a mass spectrometer, set up to investigate a stream of active particles such as atoms and radicals. The results of our investigation support the rule given in /1/ which concludes, that the primary products of ammonium perchlorate breakdown are ammonia and perchloric acid, and the expression for the first stage of breakdown has the form



Therefore, and from /1/ it follows, that the chlorine and nitric acid observed in /4/ at 230° at a pressure of 10⁻³ mm of mercury, or secondary products of the breakdown of perchloric acid and the oxidation of ammonia, which could not take place at the surface of the ammonium perchlorate as is stated in /5/, but only in the gaseous phase.

The method applied in this work can be used for the observation of active components in the impulse paralysis of other solid substances.

Results

The primary products of the breakdown reaction of ammonium perchlorate in the temperature intervals 250 - 500° are ammonia and perchloric acid.

Institute of Chemical Kinetics and Combustion, Siberia Section of the Academy of Sciences USSR received 1-11-1967.

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UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Foreign Science and Technology Center US Army Materiel Command Department of the Army		2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED	
		2b. GROUP	
3. REPORT TITLE Investigations into the Primary Products of Impulse Paralysis of Ammonium Perchlorate using a Time of Flight Mass Spectrometer			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Translation			
5. AUTHOR(S) (First name, middle initial, last name) V.V. Boldyrev, O. P. Korobeinichev and Yu. Ya. Karpenko			
6. REPORT DATE 19 September 1969		7a. TOTAL NO. OF PAGES 4	7b. NO. OF REFS N/A
8a. CONTRACT OR GRANT NO. a. PROJECT NO. 02RD500 2301 STEAP-FI		9a. ORIGINATOR'S REPORT NUMBER(S) FSTC-HT-23-490-69 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) ACSI Control Number (None)	
10. DISTRIBUTION STATEMENT This document has been approved for public release and sale; its distribution is unlimited.			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY US Army Foreign Science and Technology Center	
13. ABSTRACT The thermal breakdown of ammonium perchlorate was studied using a modified experimental method to aid in the detection of instable products. A description of the method used is described. The primary products of the breakdown reaction in the temperature range studied were amonia and perchloric acid.			

DD FORM 1473

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Security Classification

14.	KEY WORDS	LINK A		LINK B		LINK C	
		ROLE	WT	ROLE	WT	ROLE	WT
	Thermal breakdown Impulse heating Solid substance Spectrometer						

UNCLASSIFIED
Security Classification